PATENT SPECIFICATION

Inventors:—JOHN OLIVER SMITH and ALBERT EDWARD HISCOCKS.

757,233

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COMPLETE SPECIFICATION

Improvements in and relating to the Production of Regenerated Cellulose Fibres from Viscose

We, COURTAULDS LIMITED, a British Company of 16, St. Martins-le-Grand, in the City of London, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production of fibres from viscose.

In the production of fibres from viscose, viscose is normally extruded through a multi-hole jet into a coagulating bath containing an acid, usually sulphuric acid, and salts, for example sodium sulphate, zinc sulphate and magnesium sulphate, with sodium sulphate predominating. The fibres so formed are normally of substantially circular cross-section with a crenulated periphery.

It is known that by varying one or more of the spinning conditions, for example the composition of the bath, or the composition of the viscose, fibres of modified characteristics can be obtained and many proposals have been made for producing fibres having particular properties.

The object of the present invention is to produce fibres having a high bulking power, that is fibres which, for example, when twisted together will give a bulky thread.

According to the present invention a process for the production of fibres from viscose comprises extruding viscose of salt figure less than 3.5 into an aqueous coagulating bath containing less than 5 per cent by weight of sulphuric acid and more than 20 per cent by weight of salts, including at least 5 per cent of magnesium sulphate, withdrawing the fibre from the bath at a rate equal to or lower than the extrusion rate of the viscose into the bath and subsequently completing the fixation of the fibre in an aqueous acid regenerating bath having a higher concentration of acid to salt than the coagulating bath.

Preferably this regenerating bath has a ratio of salt to acid concentration less than the bisulphate ratio at which the concentration of ions is equivalent to a solution of sodium bisulphate.

The salt figure is defined as the concentration of a solution of common salt necessary just to coagulate a drop of viscose.

The preferred composition of the coagulating bath is 0.5 to 5 per cent of sulphuric acid, 10 to 25 per cent of sodium sulphate, 5 to 20 per cent of magnesium sulphate and a total salt content of about 30 per cent; the viscose preferably has a salt figure of 0.5 to 3.5 and the extrusion rate is preferably from one to twenty times the speed of withdrawal of the fibre from the bath to allow the threads to thicken on emerging from the jet.

During its passage through the coagulating bath the salts have the effect of retarding regeneration and the freshly-formed fibre is coagulated but is only partly regenerated. The fixation of the fibre is subsequently completed in the regenerating bath, for example, a dilute acid bath at a suitable temperature. The fibre is preferably passed through the regenerating bath in a relaxed condition or under low tension.

The invention also includes regenerated cellulose fibres when produced by the process according to the present invention. These fibres have a high bulking power, that is when assembled in the mass they give bulky yarns or fabrics.

When the fibres are examined under a microscope they are seen generally to have a modified cross-sectional shape as compared with normal viscose fibres. The actual shape may vary over a wide range, for example, the fibres may have a curled

[*Price* 3/-]

or crescent-shaped cross-section, or a star-shaped cross-section, or a branched cross-section.

The drawings accompanying the provisional specification, which are tracings taken from photo-micrographs, illustrate the cross-sectional form of the fibres obtained according to the present invention.

Figs. 1 to 5 show the cross-section of fibres obtained according to the invention, and

Fig. 6 shows the cross-section of fibres spun under normal conditions.

The invention illustrated by the following examples in which the percentages are by weight and all the baths are aqueous.

EXAMPLE 1

Viscose containing 7.5 per cent of cellulose, 7 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.2, was extruded at a rate of 18.6 grams per minute through a jet containing 45 holes each of 0.002 inch diameter into a coagulating bath at 55°C. containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 45 filament thread so formed was withdrawn from the bath at a rate of 40 metres per minute, the ratio of extrusion rate to take-up speed being 1:0.236, passed round a godet, through a second bath at 60°C. containing 3 per cent of sulphuric acid and a small quantity of salts carried over from the coagulating bath, over a second godet and collected in a centrifugal spinning box. The second godet was travelling at a lower speed than the first godet so that the thread was under low tension in the dilute acid bath. The cake of thread taken from the spinning box was processed in the usual way and gave a thread having a total denier of 315 (i.e., 7 denier per filament). The thread had a dry tenacity of 1.57 grams per denier and an extensibility of 18.3 per cent. Fig. 1 shows the cross-sectional shape of the fibres obtained.

EXAMPLE 2

Viscose containing 7.5 per cent of cellulose, 7.0 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 2.0 was extruded at a rate of 218 grams per minute through a jet containing 250 holes each of 0.003 inch diameter into a coagulating bath at 60°C. containing 0.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 250 filament thread so formed was withdrawn from the bath by a godet rotating at a peripheral speed of 32 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.23, passed through a bath containing 5 per cent of sulphuric acid at 30°C. and a small quantity of salts carried over from the coagulating bath, and thence to a second godet rotating at a speed such that the filaments were under low tension in the bath. From the second godet the thread passed to a cutter where it was cut into staple fibre which after processing was of 18 denier. The staple fibres had cross-sections as shown in Fig. 2.

EXAMPLE 3

Viscose containing 7.5 per cent of cellulose, 7.0 per cent of caustic soda, 2.2 per cent of combined sulphur and aged to a salt figure of 1.1 was extruded at a rate of 188 grams per minute through a jet containing 100 holes each of 0.009 inch diameter into a coagulating bath at 55°C. containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 250 filament thread so formed was withdrawn from the bath by a godet at a rate of 32 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.75, passed through a fixing bath of 6 per cent sulphuric acid and a small quantity of salts carried over from the coagulating bath at 40°C. by means of a second godet rotating at the same speed as the first godet and thence to a staple fibre cutter. The washed and dried fibres had a filament denier of 42, a dry tenacity of 1.09 grams per denier and a dry extensibility of 29.9 per cent and had a high bulking power by comparison with ordinary staple fibre of similar denier. The cross-section of the fibres was extensively branched, as shown in Fig. 3.

EXAMPLE 4

Viscose containing 7.5 per cent of cellulose, 6.5 per cent of caustic soda, 2.2 per cent of combined sulphur and aged to a salt figure of 2.3 was extruded at a rate of 18 grams per minute through a jet containing 20 holes each of 0.002 inch diameter into a coagulating bath at 60°C. containing 3.5 per cent of sulphuric acid, 10 per cent of magnesium sulphate and 20 per cent of sodium sulphate. The 20 filament thread was withdrawn from the bath at a speed of 40 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.1 and collected in a spinning box. The thread from the cake was reeled, the skeins were fixed in 2 per cent sulphuric acid at 30°C. and washed and dried. The dried thread had a tenacity of 1.07 grams per denier and an extensibility of 31.1 per cent. The filaments were star-shaped in cross-section as shown in Fig. 4.

EXAMPLE 5

Viscose containing 7.5 per cent of cellulose, 6.5 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.7, was extruded at a rate

of 199 grams per minute through a jet containing 1200 holes each of 0.002 inch diameter into a coagulating bath containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 1200 filament thread so formed was withdrawn from the bath by a godet rotating at a peripheral speed of 32 metres per minute, passed through a fixing bath of .2 per cent sulphuric acid and a small quantity of salts carried over from the coagulating bath at 40°C. over a second godet rotating at the same speed as the first godet and thence to a staple fibre cutter. The washed and dried staple fibre had a high bulking power compared with ordinary staple fibre and had a filament denier of 4.9. The dry tenacity was 0.93 grams per denier and the dry extensibility 32.5 per cent. In cross-section, the fibres were markedly branched, as shown in Fig. 5.

EXAMPLE 6

Viscose containing 8.3 per cent of cellulose, 6.0 per cent soda and 2.3 per cent of combined sulphur and aged to a salt figure of 1.7 was extruded at a rate of 21.5 grams per minute through a jet containing 20 holes each of 0.002 inch diameter into a coagulating bath at 55°C. containing 1 per cent of sulphuric acid, 20 per cent of magnesium sulphate and 10 per cent of sodium sulphate. The 20 filament thread was withdrawn at a rate of 40 metres per minute, the ratio of extrusion rate to take-up speed being 1:0.083, passed round a godet and was collected in a centrifugal spinning box. The thread so formed was wound into skeins which were fixed in a bath containing 2 per cent of sulphuric acid at 30°C. washed and dried. The filaments in the threads were each of 24 denier and had a cross-sectional shape similar to those shown in Figs. 1 and 2.

EXAMPLE 7

Viscose containing 7.5 per cent cellulose, 6.0 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.1 was extruded at a rate of 136 grams per minute through a jet containing 100 holes each of 0.003 inch diameter into a coagulating bath at 55°C. containing 1 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 100 filament thread was withdrawn at a rate of 32 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.083 and passed under normal tension through a bath containing 3 per cent of sulphuric acid and a small quantity of salts carried over from the coagulating bath at 50°C. to complete this fixation. The thread was then cut into staple fibre and processed in the usual way to give staple fibres of 50 denier with a cross-sectional shape similar to those shown in Figs. 1 and 2.

What we claim is:—

1. A process for the production of fibres from viscose comprising extruding viscose of salt figure less than 3.5 into an aqueous coagulating bath containing less than 5 per cent by weight of sulphuric acid and more than 20 per cent by weight of salt 3, including at least 5 per cent of magnesium sulphate, withdrawing the fibre from the bath at a rate equal to or lower than the extrusion rate of the viscose into the bath and subsequently completing the fixation of the fibre in an aqueous acid bath having a higher concentration of acid to salt than the coagulating bath.

2. A process as claimed in Claim 1, wherein the coagulating bath contains 0.5 to 5 per cent of sulphuric acid, 10 to 25 per cent of sodium sulphate and 5 to 20 per cent of magnesium sulphate with a total salt content of about 30 per cent.

3. A process as claimed in Claim 1 or 2, wherein the viscose has a salt figure of 0.5 to 3.5.

4. A process as claimed in Claim 1, 2 or 3, wherein the extrusion rate of the viscose is from one to twenty times the speed of withdrawal of the fibre from the bath.

5. A process as claimed in any of Claims 1 to 4, wherein the fixation of the fibre is completed in a relaxed condition or under low tension.

6. A process as claimed in Claim 1 substantially as described in any of the foregoing Examples.

7. Regenerated cellulose fibres having a high bulking power when produced by a process as claimed in any of the preceding claims.

J. Y. & G. W. JOHNSON,
47, Lincoln's Inn Fields, London, W.C.2.
Chartered Patent Agents.

PROVISIONAL SPECIFICATION

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It is known that by varying one or more of the spinning conditions, for example the composition of the bath, or the composition of the viscose, fibres of modified characteristics can be obtained and many proposals have been made for producing fibres having particular properties.

The object of the present invention is to produce fibres having a high bulking power, that is fibres which, for example, when twisted together will give a bulky thread.

According to the present invention a process for the production of fibres from viscose comprises extruding viscose of low salt figure into a coagulating bath containing a low concentration of acid and a high concentration of salts, including at least 5 per cent of magnesium sulphate, withdrawing the fibre from the bath at a rate equal to or lower than the extrusion rate of the viscose into the bath and subsequently fixing the fibre in an acid bath.

The preferred composition of the coagulating bath is 0.5 to 5 per cent of sulphuric acid, 10 to 25 per cent of sodium sulphate, 5 to 20 per cent of magnesium sulphate and a total salt content of about 30 per cent; the viscose preferably has a salt figure of 0.5 to 3.5 and the extrusion rate is preferably from one to twenty times the take-up speed.

During its passage through the coagulating bath the freshly-formed fibre is coagulated but is either not regenerated at all or is only partly regenerated and the fibre is subsequently fixed in the regenerating bath, for example, a dilute acid bath at a suitable temperature. The fibre is preferably passed through the regenerating bath in a relaxed condition or under low tension.

The invention also includes regenerated cellulose fibres when produced by the process according to the present invention. These fibres have a high bulking power, that is when assembled in the mass they give bulky yarns or fabrics.

When the fibres are examined under a microscope they are seen generally to have a modified cross-sectional shape as compared with normal viscose fibres. The actual shape may vary over a wide range, for example, the fibres may have a curled or crescent-shaped cross-section, or a star-shaped cross-section, or a branched cross-section.

The accompanying drawings, which are tracings taken from photo-micrographs, illustrate the cross-sectional form of the fibres obtained according to the present invention.

Figs. 1 to 5 show the cross-section of fibres obtained according to the invention, and .

Fig. 6 shows the cross-section of fibres spun under normal conditions.

The invention is illustrated by the following Examples in which the percentages are by weight.

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Viscose containing 7.5 per cent of cellulose, 7 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.2 was extruded at a rate of 18.6 grams per minute through a jet containing 45 holes each of 0.002 inch diameter into a coagulating bath at 55°C. containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 45 filament thread so formed was withdrawn from the bath at a rate of 40 metres per minute, the ratio of extrusion rate to take-up speed being 1:0.236, passed round a godet, through a second bath at 60°C containing 3 per cent of sulphuric acid, over a second godet and collected in a centrifugal spinning box. The second godet was travelling at a lower speed than the first godet so that the thread was under low tension in the dilute acid bath. The cake of thread taken from the spinning box was processed in the usual way and gave a thread having a total denier of 315 (i.e. 7 denier per filament). The thread had a dry tenacity of 1.57 grams per denier and an extensibility of 18.3 per cent. Fig. 1 shows the cross-sectional shape of the fibres obtained.

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under low tension in the bath. From the second godet the thread passed to a cutter where it was cut into staple fibre which after processing was of 18 denier. The staple fibres had cross-sections as shown in Fig. 2.

EXAMPLE 3

Viscose containing 7.5 per cent of cellulose, 7 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.1 was extruded at a rate of 188 grams per minute through a jet containing 100 holes each of 0.009 inch diameter into a coagulating bath at 55°C. containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 250 filament thread so formed was withdrawn from the bath by a godet at a rate of 32 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.75, passed through a fixing bath of 6 per cent sulphuric acid at 40°C. by means of a second godet rotating at the same speed as the first godet and thence to a staple fibre cutter. The washed and dried fibres had a filament denier of 42, a dry tenacity of 1.09 grams per denier and a dry extensibility of 29.9 per cent and had a high bulking power by comparison with ordinary staple fibre of similar denier. The cross-section of the fibres was extensively branched, as shown in Fig. 3.

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Viscose containing 7.5 per cent of cellulose, 6.5 per cent of caustic soda, 2.2 per cent of combined sulphur and aged to a salt figure of 2.3 was extruded at a rate of 18 grams per minute through a jet containing 20 holes each of 0.002 inch diameter into a coagulating bath at 60°C. containing 3.5 per cent of sulphuric acid, 10 per cent of magnesium sulphate and 20 per cent of sodium sulphate. The 20 filament thread was withdrawn from the bath at a speed of 40 metres per minute to give a ratio of extrusion rate to take-up speed of 1:0.1 and collected in a spinning box. The thread from the cake was reeled, the skeins were fixed in 2 per cent sulphuric acid at 30°C. and washed and dried. The dried thread had a tenacity of 1.07 grams per denier and an extensibility of 31.1 per cent. The filaments were star-shaped in cross-section as shown in Fig. 4.

EXAMPLE 5

Viscose containing 7.5 per cent of cellulose, 6.5 per cent of caustic soda and 2.2 per cent of combined sulphur and aged to a salt figure of 1.7, was extruded at a rate of 199 grams per minute through a jet containing 1200 holes each of 0.002 inch diameter into a coagulating bath containing 1.5 per cent of sulphuric acid, 15 per cent of magnesium sulphate and 15 per cent of sodium sulphate. The 1200 filament thread so formed was withdrawn from the bath by a godet rotating at a peripheral speed of 32 metres per minute, passed through a fixing bath of 2 per cent sulphuric acid at 40°C., over a second godet rotating at the same speed as the first godet and thence to a staple fibre cutter. The washed and dried staple fibre had a high bulking power compared with ordinary staple fibre and had a filament denier of 4.9. The dry tenacity was 0.93 grams per denier and the dry extensibility 32.5 per cent. In cross-section, the fibres were markedly branched, as shown in Fig. 5.

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Viscose containing 8.3 per cent of cellulose, 6.0 per cent soda and 2.3 per cent of combined sulphur and aged to a salt figure of 1.7 was extruded at a rate of 21.5 grams per minute through a jet containing 20 holes each of 0.002 inch diameter into a coagulating bath at 55°C. containing 1 per cent of sulphuric acid, 20 per cent of magnesium sulphate and 10 per cent of sodium sulphate. The 20 filament thread was withdrawn at a rate of 40 metres per minute, the ratio of extrusion rate to take-up speed being 1:0.083, passed round a godet and was collected in a centrifugal spinning box. The thread so formed was wound into skeins which were fixed in a bath containing 2 per cent of sulphuric acid at 30°C. washed and dried. The filaments in the threads were each of 24 denier and had a cross-sectional shape similar to those shown in Figs. 1 and 2.

EXAMPLE 7

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J. Y. & G. W. JOHNSON,
47, Lincoln's Inn Fields, London, W.C.2.
Chartered Patent Agents.

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757,233 PROVISIONAL SPECIFICATION
1 SHEET

This drawing is a reproduction of the Original on a reduced scale.

FIG.1.

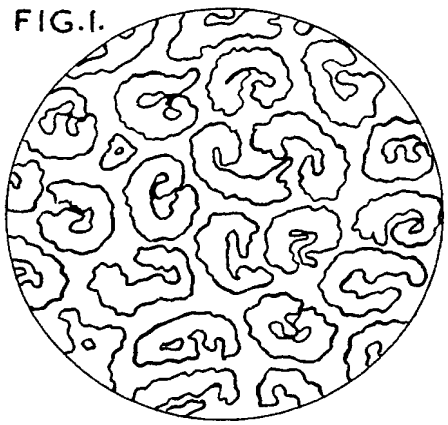

FIG.2.

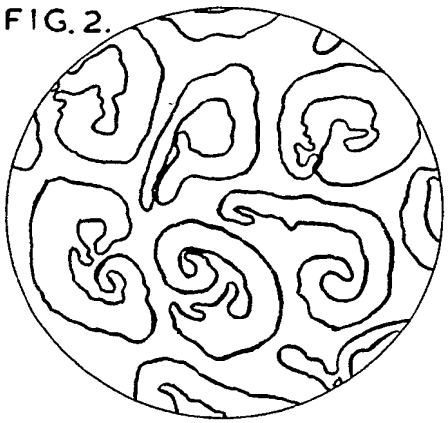

FIG.3.

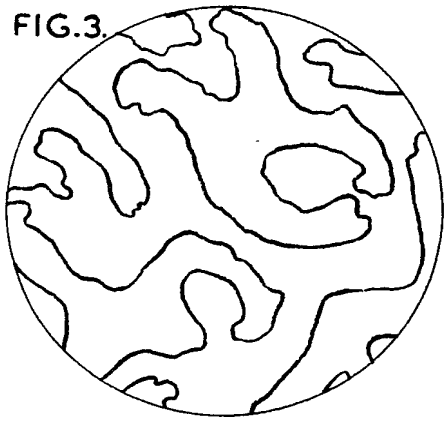

FIG.4.

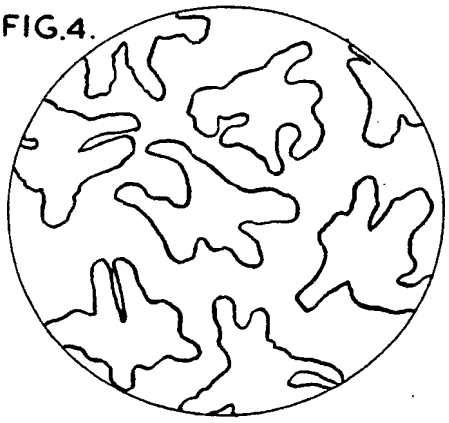

FIG.5.

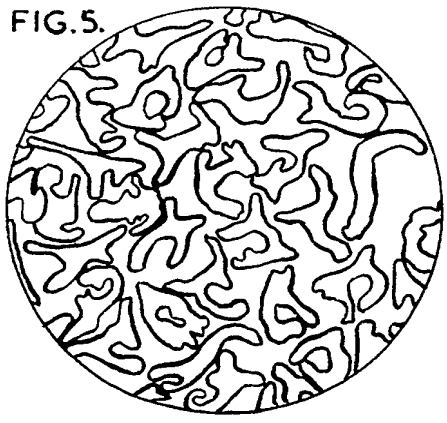

FIG.6.